

# PATENT ABSTRACTS OF JAPAN

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## (54) FIBROUS PREFORM CONTAINING CYCLIC POLYESTER OLIGOMER AND ITS PRODUCTION

(57)Abstract:

**PURPOSE:** To provide a shapable article containing a fiber mat impregnated with a specific oligomer compsn., excellent in shape holdability and bonding holdability at a time of treatment operation before molding and useful for various thermoplastic composite articles.

**CONSTITUTION:** The objective shapable article contains a fiber mat (e.g. carbon fiber mat or glass fiber mat) obtained by impregnating the fiber mat with a shape stabilizing effective amt. up to 10 wt.% of a macrocyclic poly(alkylene dicarboxylate) oligomer compsn. [e.g.; poly(1,4-butyreneterephalate) oligomer compsn.] not containing a polymerizing catalyst of a macrocyclic poly(alkylenedicarboxylate) oligomer compsn. and a linear polyester.

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**CLAIMS**

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**[Claim(s)]**

[Claim 1]A shape stable effective dose of at least a kind of macro cyclic poly (alkylene dicarboxylate) oligomer constituent which does not contain linear polyester substantially and does not include substantially a catalyst for a polymerization of a macro cyclic poly (alkylene dicarboxylate) oligomer constituent, namely, an article which contains quantity up to about 10 % of the weight, and at least one textiles mat come out and impregnated based on weight of a textiles mat and which can be modeled.

[Claim 2]The article according to claim 1 in which these textiles are carbon, glass, polyamide by which orientation was carried out highly, or a boron fiber.

[Claim 3]The article according to claim 2 in which these textiles are glass fiber.

[Claim 4]The article according to claim 2 in which this macro cyclic oligomer constituent is a mixture of oligomer.

[Claim 5]The article according to claim 4 in which a rate of a linear polyester kind in this macro cyclic oligomer constituent does not exceed about 0.1 % of the weight.

[Claim 6]The article according to claim 4 in which this macro cyclic oligomer is poly (ethylene terephthalate), poly (1,4-butylene terephthalate), poly (ethylene isophthalate), poly (1,4-butylene isophthalate) oligomer, or those mixtures.

[Claim 7]The article according to claim 6 in which this macro cyclic oligomer is poly (1, 4-butylene terephthalate) oligomer.

[Claim 8]The article according to claim 7 a rate of a macro cyclic oligomer constituent is [ article ] to about 5 % of the weight based on weight of this textiles mat.

[Claim 9]A shape stable effective dose of at least a kind of macro cyclic poly (alkylene dicarboxylate) oligomer constituent which does not contain linear polyester for a textiles mat substantially, and does not include substantially a catalyst for a polymerization of a macro cyclic poly (alkylene dicarboxylate) oligomer constituent, namely, a method of stabilizing

quantity up to about 10 % of the weight, and shape of at least one textiles mat which comes out and includes a process of being impregnated based on weight of a textiles mat.

[Claim 10]A way according to claim 9 these textiles are carbon, glass, polyamide by which orientation was carried out highly, or a boron fiber.

[Claim 11]A way according to claim 10 these textiles are glass fiber.

[Claim 12]A way according to claim 11 this macro cyclic oligomer constituent is a mixture of oligomer.

[Claim 13]A way according to claim 12 a rate of a linear polyester kind in this macro cyclic oligomer constituent does not exceed about 0.1 % of the weight.

[Claim 14]A way according to claim 13 this macro cyclic oligomer is poly (ethylene terephthalate), poly (1,4-butylene terephthalate), poly (ethylene isophthalate), poly (1,4-butylene isophthalate) oligomer, or those mixtures.

[Claim 15]A way according to claim 14 this macro cyclic oligomer is poly (1, 4-butylene terephthalate) oligomer.

[Claim 16]A way according to claim 15 a rate of a macro cyclic oligomer constituent is to about 5 % of the weight based on weight of this textiles mat.

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**DETAILED DESCRIPTION**

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**[Detailed Description of the Invention]****[0001]**

[Industrial Application]This invention relates to the preforming thing for \*\*\*\*\* complexes (preform) especially about the modeled polymer complex.

**[0002]**

[Description of the Prior Art]the fiber products held stably at the convenient specific outline for the solidification (solidification) to the complex products in which the fiber preforming thing was completed -- typical -- a mat -- it comes out. The outline and thickness are equivalent to the outline and thickness which the usually completed product is expected. Preferably, this preforming thing is further characterized in that an excessive swelling and solution (\*\*\*\*) \*\*\*\* textiles are the minimum. This preforming thing is often manufactured as a roll of a substance similar to textiles (fabric) at first. After modeling this substance, and combining with resin or other substances for being impregnated and solidifying in a press, autoclave, or a metallic mold, the complex products which made harden this resin and were completed can be formed.

[0003]This preforming thing should be easily processed without damage, and should be put on typical ambient environment, and should be structurally stable. There are other desirable standards by a low labor cost, prompt manufacture, and minimum-ization of waste.

**[0004]**

[Problem(s) to be Solved by the Invention]One of the main problems in manufacture of a preforming thing is guaranteeing a fiber reinforcing member's suitable arrangement and unity (integrity). The bulking agent used into glass or a complex like a graphite fiber is not flexible (drapable). That is, these textiles will always return to the original shape of them, when it changes elastically and \*\* and others also removes flow stress. These textiles have the difficulty of making insufficient the adhesiveness between layers like [ again / between the

portion by which the textiles mat was woven, and a non-\*\*\*\*ing portion ] (tack), i.e., an adhesive property, (adhesion). As the result, lamination (lay-up) of textiles may serve as time and a \*\*\*\*\* process of expense, and there is a possibility that achievement of arrangement of the complex of good unity may become difficult or impossible.

[0005]In order to carry out fabricating to the complex products which had the fiber mat modeled, generally three art has been used. The first art cuts this mat in only suitable shape, and is arranging it in a metallic mold or other shaping tools. This method is useful for the products of the even or shape very near it. The second art is using the operation for formation of a braid (braiding) and a three-dimensional weaving structure or manufacture of a mat like a suture (stitching), and an assembly (assembly). \*\*\*\*\* operation can only be applied only to the structure of the only limited range, and, moreover, will be able to become labor-intensive.

[0006]In order to hold the unity of a preforming thing so that it may be indicated by the U.S. Pat. No. 5,071,711 specification, for example, a binding material (binder) is used for the third art. Generally a substance useful as a binding material has the adhesive property limited at least. These binding material substances hold this preforming thing together during the processing, and before they arrange this preforming thing in a metallic mold by it, they may be made to hold it in desired shape, when using it for a mat.

[0007]Now, many weaving or a non-\*\*\*\*ing mat may be obtained with the already used binding material. Typically, use of the binding material to nonsequential textiles collects these textiles on a screen by vacuum suction operation, and is attained by spraying the solution or slurry of a binding material on it. Operation like spraying can also attain the use to continuous textiles.

[0008]However, the binding material used commercially may not suit no formation method of complexes. For example, a U.S. Pat. No. 5,191,013 specification is impregnated by macro cyclic poly (alkylene dicarboxylate) oligomer in a bulking agent, Subsequently, although the method of manufacturing an article by carrying out ring opening polymerization of this cyclic oligomer is indicated, this polymerization is often performed at 200 \*\* or the temperature beyond it, and many binding material substances by which normal use is carried out cannot bear this temperature. And use of an unsuitable binding material has a possibility of causing a reaction harmful between this binding material and this resinic body substance, and, as a result, brings about change of the character of one side of them, or both.

[0009]

[Means for Solving the Problem]This invention can hold those shape and unity during repetitive manufacture before shaping, and treating operation as a fiber preforming thing, and provides a fiber preforming thing which is what can moreover be fabricated on thermoplastic complex articles by various methods. This invention provides a method of giving dimensional stability to a bulking agent of a gestalt of a preforming thing.

[0010]Therefore, according to one gestalt of this invention. A shape stable effective dose of at

least a kind of macro cyclic poly (alkylene dicarboxylate) oligomer constituent which does not contain linear polyester substantially and does not include substantially a catalyst for a polymerization of a macro cyclic poly (alkylene dicarboxylate) oligomer constituent, that is, based on weight of a textiles mat, quantity up to about 10 % of the weight and an article which comes out and contains at least one impregnated textiles mat and which can be modeled are provided.

[0011]

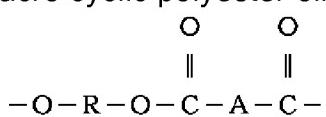
[Example]One of the important character of the article of this invention is a point which is what they can model. That is, arbitrary \*\*\*\*\* articles can be modeled in specific shape, and it may be the plane or more complicated shape a room temperature or after heating. At one process or another process, it is shape approximated to the shape which the usually completed complex article is expected.

[0012]The textiles mat which may be used in this invention may contain the known arbitrary substances for fiber reinforcement in the art concerned. Both and those mixtures of the shredded textiles (chopped fiber) and a continuous fiber can be used. A suitable substance includes carbon, glass, the polyamide for your kind consideration, and boron. Carbon fiber and glass fiber are often preferred.

[0013]This substance for reinforcement may be a gestalt of a single layer or two or more layers. In order to manufacture a multilayer article, these layers are accumulated under the conditions which may make those shape hold in a metallic mold or the device for modeling equivalent to it typically. The substance (it may be hereafter called a "binding material") for giving dimensional stability according to this invention is a kind of united type macroscopic cyclic poly (alkylene dicarboxylate) (it may only be hereafter called "polyester") oligomer constituent at least. The term "macroscopic cyclic one" used in this specification means existence of a synthetic (overall) ring structure as the arbitrary alicycle fellows, the aromatic series, or the heterocyclic ring which exists there independently.

[0014]All of the mixture of a single compound and a compound can be used. These macro cyclic compounds can be monomers-like, i.e., they may include a single structural unit. However, in many cases, the mixture of a kind of macro cyclic oligomer and macro cyclic oligomer of desirable various degrees of polymerization is used at least. A \*\*\*\*\* mixture contains the oligomer which usually has 2 thru/or about 12 degree of polymerization at a main rate.

[0015]These macro cyclic polyester oligomers are formulas. :



The structural unit of (the inside of a formula and R are alkynes, mono-, or polyoxyalkylene

groups including the straight chain of about two to eight atoms, and A is m-, the monocyclic aromatic compound by which the p bond was carried out, or an alicycle fellows group) is included. \*\*\*\*\* oligomer can be manufactured so that it may be indicated by U.S. Pat. No. 5,039,783, 5,191,013, and the 5,231,161 specification. Preferably, R is ethylene or (especially) a 1,4-butylene group, and A is m-phenylene or (especially) p-phenylene group.

[0016]Therefore, desirable oligomer is poly (ethylene terephthalate), poly (1, 4-butylene terephthalate), or corresponding oligomer of isophthalate. The mixture of these molecular species is also included. Especially poly (1, 4-butylene terephthalate) oligomer is preferred. Neither of macro cyclic polyester oligomer used according to this invention contains substantially the linear polyester which includes a line polyester oligomer and the amount polyester of polymers. When it obtains with it and a small percentage also exists, by the time linear polyester is inconvenient, it will increase the viscosity of this macro cyclic constituent, corresponds to it and reduces the ease of handling of this constituent under formation of a preforming thing. Generally, the linear polyester kind (oligomer and the amount polymer of polymers) should not exist at a rate exceeding about 2 % of the weight, and should not exceed about 0.1 % of the weight preferably.

[0017]It is required that a macro cyclic oligomer constituent should not contain the catalyst of \*\* for conversion of the object for a polymerization, i.e., linear polyester. The operation which the oligomer constituent in a preforming thing means stabilizes it, and this is making the processing easy and is true for the reason for not being converted into a linear polymer. Arbitrary catalysts required for eye others even in such a case are not under manufacture of a preforming thing, and processing, \*\*\*\*\* conversion may occur in the latter part more so that it may mention later, but it is not and they should be rather added in down stream processing after it.

[0018]The article of this invention can be manufactured by the method of including conventional modeling operation. The modeling can attain processing with manufacture and the roll of a preforming thing, processing in a metallic mold, or the processing on a mandrel in various stages which the use front stirrup of a macro cyclic polyester composition performs simultaneously with use. This constituent can be used with the gestalt of a solid or a fluid. When it is used as a solid, it should be dissolved by it by heating a preforming thing.

[0019]Use operation suitable for a solid constituent includes spraying (SUPURINKURU use) and pouring (pouring). Brush coating, roller coating, immersion coating, flow coating, spraying coating, etc. can be used for use of a fluid. It is not required to impregnate textiles with a binding material. Although the use to the surface is usually enough, a certain amount of osmosis can also be permitted.

[0020]A binding material is used in a dimensional stabilization effective dose. That is, the article of this invention should give it dimensional stability, and should contain only the binding

material which is sufficient for holding the unity. In any cases, the binding material of the rate which exceeds about 10 % of the weight based on the weight of a mat shall not use it, and, in many cases, use of the binding material up to about 5 % of the weight is enough as it.

[0021]So, manufacture of prepreg (prepregs= resin osmosis processing material) is not meant. About prepreg, the time and effort for solidifying resin in proportion of the capacity corresponding to the capacity expected by the last article is required, eliminating the drawn air or other gas, or preventing to the minimum. \*\*\*\*\* solidification is not [ that it is not required, either or ] again especially desirable in manufacture of a preforming thing, either. It is because solidification will take place innately between the last molding operation.

[0022]This preforming thing can be further processed into a person skilled in the art by a known method following formation of a preforming thing. For example, if this preforming thing is necessary, it can solidify in a press or autoclave, or \*\*\*\*\* (stamping) of it can be carried out in a metallic mold. Converting the article of this invention into prepreg or a resinic body complex by addition of resin of the amount of another further is also meant. This conversion can be attained using operation like transfer molding of resin. The arbitrary thermoplastics which includes another macro cyclic polyester for this purpose can be used under existence of the catalyst for the conversion to linear polyester of that. If it is necessary, another modeling operation can also be used in advance of shaping.

[0023]Another gestalt of this invention a textiles mat at the time mentioned above The shape stable effective dose of at least a kind of macro cyclic polyester oligomer constituent, that is, it is the method of stabilizing the quantity up to about 10 % of the weight, and the shape of at least one textiles mat which comes out and includes the process of being impregnated based on the weight of a textiles mat. the next -- this invention -- linear polyester (; which includes oligomer -- these by processing with silica gel.) And it was removed, the example which uses the macro cyclic poly (1, 4-butylene terephthalate) oligomer constituent which mainly has 2 thru/or about 12 degree of polymerization without containing a catalyst explains. The test piece with the size of a 17.8-cm angle and the weight of about 9.6 g of glass tissue was placed on the aluminium sheet, and 390 mg (it is based on a mat and is 4 % of the weight) of macro cyclic oligomer was distributed to one whole field of this test piece carefully and uniformly. The second cloth test piece was manufactured similarly, and it has arranged on the first cloth test piece. The assembly obtained was inserted in the decompression bag made of nylon, and was sealed, and it compressed under the vacuum, and it was inserted in a 190 \*\* heat convection furnace after that. At this temperature, this macro cyclic oligomer constituent had the viscosity of about 40 sufficiently low centipoises, although applied.

[0024]60 minutes afterward, having been dissolved and applied was admited, the preforming thing which might be written was picked out from the furnace, and it cooled to the room temperature under the vacuum, and this resin was picked out from the bag. This preforming

thing showed good solidification, and supported the weight of itself, and had sufficient rigidity to bear repetitive \*\*\*\*\* (repeated striking) to the upper surface (countertop).

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## TECHNICAL FIELD

[Industrial Application] This invention relates to the preforming thing for \*\*\*\*\* complexes (preform) especially about the modeled polymer complex.

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## PRIOR ART

[Description of the Prior Art]the fiber products held stably at the convenient specific outline for the solidification (solidification) to the complex products in which the fiber preforming thing was completed -- typical -- a mat -- it comes out. The outline and thickness are equivalent to the outline and thickness which the usually completed product is expected. Preferably, this preforming thing is further characterized in that an excessive swelling and solution (\*\*\*\*) \*\*\*\* textiles are the minimum. This preforming thing is often manufactured as a roll of a substance similar to textiles (fabric) at first. After modeling this substance, and combining with resin or other substances for being impregnated and solidifying in a press, autoclave, or a metallic mold, the complex products which made harden this resin and were completed can be formed.

[0003]This performing thing should be easily processed without damage, and should be put on typical ambient environment, and should be structurally stable. There are other desirable standards by a low labor cost, prompt manufacture, and minimum-ization of waste.

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**TECHNICAL PROBLEM**

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[Problem(s) to be Solved by the Invention] One of the main problems in manufacture of a preforming thing is guaranteeing a fiber reinforcing member's suitable arrangement and unity (integrity). The bulking agent used into glass or a complex like a graphite fiber is not flexible (drapable). That is, these textiles will always return to the original shape of them, when it changes elastically and \*\* and others also removes flow stress. These textiles have the difficulty of making insufficient the adhesiveness between layers like [ again / between the portion by which the textiles mat was woven, and a non-\*\*\*\*ing portion ] (tack), i.e., an adhesive property, (adhesion). As the result, lamination (lay-up) of textiles may serve as time and a \*\*\*\*\* process of expense, and there is a possibility that achievement of arrangement of the complex of good unity may become difficult or impossible.

[0005] In order to carry out fabricating to the complex products which had the fiber mat modeled, generally three art has been used. The first art cuts this mat in only suitable shape, and is arranging it in a metallic mold or other shaping tools. This method is useful for the products of the even or shape very near it. The second art is using the operation for formation of a braid (braiding) and a three-dimensional weaving structure or manufacture of a mat like a suture (stitching), and an assembly (assembly). \*\*\*\*\* operation can only be applied only to the structure of the only limited range, and, moreover, will be able to become labor-intensive.

[0006] In order to hold the unity of a preforming thing so that it may be indicated by the U.S. Pat. No. 5,071,711 specification, for example, a binding material (binder) is used for the third art. Generally a substance useful as a binding material has the adhesive property limited at least. These binding material substances hold this preforming thing together during the processing, and before they arrange this preforming thing in a metallic mold by it, they may be made to hold it in desired shape, when using it for a mat.

[0007] Now, many weaving or a non-\*\*\*\*ing mat may be obtained with the already used binding material. Typically, use of the binding material to nonsequential textiles collects these textiles

on a screen by vacuum suction operation, and is attained by spraying the solution or slurry of a binding material on it. Operation like spraying can also attain the use to continuous textiles. [0008]However, the binding material used commercially may not suit no formation method of complexes. For example, a U.S. Pat. No. 5,191,013 specification is impregnated by macro cyclic poly (alkylene dicarboxylate) oligomer in a bulking agent, Subsequently, although the method of manufacturing an article by carrying out ring opening polymerization of this cyclic oligomer is indicated, this polymerization is often performed at 200 \*\* or the temperature beyond it, and many binding material substances by which normal use is carried out cannot bear this temperature. And use of an unsuitable binding material has a possibility of causing a reaction harmful between this binding material and this resinic body substance, and, as a result, brings about change of the character of one side of them, or both.

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## MEANS

[Means for Solving the Problem] This invention can hold those shape and unity during repetitive manufacture before shaping, and treating operation as a fiber preforming thing, and provides a fiber preforming thing which is what can moreover be fabricated on thermoplastic complex articles by various methods. This invention provides a method of giving dimensional stability to a bulking agent of a gestalt of a preforming thing.

[0010]Therefore, according to one gestalt of this invention. A shape stable effective dose of at least a kind of macro cyclic poly (alkylene dicarboxylate) oligomer constituent which does not contain linear polyester substantially and does not include substantially a catalyst for a polymerization of a macro cyclic poly (alkylene dicarboxylate) oligomer constituent, that is, based on weight of a textiles mat, quantity up to about 10 % of the weight and an article which comes out and contains at least one impregnated textiles mat and which can be modeled are provided.

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**EXAMPLE**

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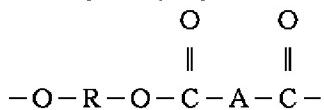
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[0014]All of the mixture of a single compound and a compound can be used. These macro cyclic compounds can be monomers-like, i.e., they may include a single structural unit. However, in many cases, the mixture of a kind of macro cyclic oligomer and macro cyclic oligomer of desirable various degrees of polymerization is used at least. A \*\*\*\*\* mixture contains the oligomer which usually has 2 thru/or about 12 degree of polymerization at a main rate.

[0015]These macro cyclic polyester oligomers are formulas. :



The structural unit of (the inside of a formula and R are alkynes, mono-, or polyoxyalkylene groups including the straight chain of about two to eight atoms, and A is m-, the monocyclic aromatic compound by which the p bond was carried out, or an alicycle fellows group) is included. \*\*\*\*\* oligomer can be manufactured so that it may be indicated by U.S. Pat. No. 5,039,783, 5,191,013, and the 5,231,161 specification. Preferably, R is ethylene or (especially) a 1,4-butylene group, and A is m-phenylene or (especially) p-phenylene group.

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[0023]Another gestalt of this invention a textiles mat at the time mentioned above The shape stable effective dose of at least a kind of macro cyclic polyester oligomer constituent, that is, it is the method of stabilizing the quantity up to about 10 % of the weight, and the shape of at least one textiles mat which comes out and includes the process of being impregnated based on the weight of a textiles mat. the next -- this invention -- linear polyester (; which includes oligomer -- these by processing with silica gel.) And it was removed, the example which uses the macro cyclic poly (1, 4-butylene terephthalate) oligomer constituent which mainly has 2 thru/or about 12 degree of polymerization without containing a catalyst explains. The test piece with the size of a 17.8-cm angle and the weight of about 9.6 g of glass tissue was placed on the aluminium sheet, and 390 mg (it is based on a mat and is 4 % of the weight) of macro cyclic oligomer was distributed to one whole field of this test piece carefully and uniformly. The second cloth test piece was manufactured similarly, and it has arranged on the first cloth test piece. The assembly obtained was inserted in the decompression bag made of nylon, and was sealed, and it compressed under the vacuum, and it was inserted in in a 190 \*\* heat

convection furnace after that. At this temperature, this macro cyclic oligomer constituent had the viscosity of about 40 sufficiently low centipoises, although applied.

[0024]60 minutes afterward, having been dissolved and applied was admited, the preforming thing which might be written was picked out from the furnace, and it cooled to the room temperature under the vacuum, and this resin was picked out from the bag. This preforming thing showed good solidification, and supported the weight of itself, and had sufficient rigidity to bear repetitive \*\*\*\*\* (repeated striking) to the upper surface (countertop).

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**DETAILED DESCRIPTION**

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**[Detailed Description of the Invention]****[0001]**

[Field of the Invention]This invention relates to the rolled letter paper for tobacco for manufacturing tobacco with few sidestream smokes.

**[0002]**

[Description of the Prior Art]If the mainstream smoke attracted by a smoker through a tobacco main part and fire leave just on the occasion of smoking of tobacco, the sidestream smoke which rises from this tip will occur, but a sidestream smoke also causes a health issue at the same time it gives the surrounding nonsmoker displeasure. For this reason, it ranks with not affecting smoking taste, having the moderate rate of combustion, that the leaf of a cigarette is transparent and is not seen (opacity), self-winding fitness (tensile strength), etc. as the required characteristic of the rolled letter paper for tobacco, and it is becoming still more important to decrease a sidestream smoke in recent years.

[0003]About decreasing the sidestream smoke of tobacco, conventionally Then, JP,48-9000,A, JP,48-61699,A, JP,58-183082,A, JP,63-87967,A, JP,2-99699,A, JP,2-501075,A, JP,3-33299,A, JP,3-43068,A, JP,3-151868,A, Many methods are proposed by gazettes, such as JP,3-180597,A, JP,3-500553,A, JP,5-236929,A, JP,5-324397,A, JP,10-81,A, and JP,11-124800,A.

[0004]For example, to JP,5-236929,A, in order to decrease a sidestream smoke, making the rolled letter paper for tobacco contain carbon is indicated.

In JP,10-81,A, in order to decrease a sidestream smoke, the rolled letter paper for tobacco using pulp of the high degree of beating is indicated.

To JP,11-124800,A, in order to mask the bad smell of a sidestream smoke, making lactone contain is indicated. although that which surely is effective is accepted, a manufacturing method is difficult, these proposals have many problems, impairing the smoking taste of

tobacco or becoming a cost hike, and it is actual that the yet sufficiently satisfying rolled letter paper for tobacco is not found out.

[0005]By using alkali metal salt especially potassium citrate, and sodium acid citrate for the rolled letter paper for tobacco as a combustion promotion additive, it is known that a sidestream smoke will decrease and, generally it is used (U.S. Pat. No. 4321377 specification). However, although it is possible for the rate of combustion to become quick and to decrease a sidestream smoke to some extent as a result by adding alkali metal salt, there is evil in which regulation is difficult, the rate of combustion will fall rather if alkali metal salt is added superfluously, or smoking taste falls. Therefore, it is difficult to decrease a sidestream smoke, without changing smoking taste only by addition of alkali metal salt.

[0006]

[Problem(s) to be Solved by the Invention]An object of this invention is to provide the rolled letter paper for tobacco which decreased in number the sidestream smoke remarkably as compared with the conventional rolled letter paper for tobacco, not affecting the smoking taste of tobacco but holding the moderate rate of combustion.

[0007]

[Means for Solving the Problem]A result of this invention persons having investigated a new combustion promotion additive, and having considered application to rolled letter paper for tobacco, Without changing an effect which cannot be attained if the above-mentioned alkali metal salt was only used as a combustion promotion additive, i.e., smoking taste, a substance to which a sidestream smoke is reduced is found out and it came to complete this invention.

[0008]This invention for solving the above-mentioned technical problem includes the following inventions.

(1) Rolled letter paper for tobacco using pulp and precipitated calcium carbonate as the main ingredients, and containing goethite further.

(2) Rolled letter paper for tobacco which contains goethite (alpha-FeOOH) of 0.1 to 20 mass part to bone-dry pulp 100 mass part in rolled letter paper for tobacco which uses pulp and precipitated calcium carbonate as the main ingredients.

[0009](3) (1) which contains an alkali-metal-salt combustion improver of 0.5 - 5.0 mass part to bone-dry pulp 100 mass part further, or rolled letter paper for tobacco given in (2) paragraphs.

(4) Rolled letter paper for tobacco given in any 1 paragraph of a (1) paragraph - (3) paragraph whose permeability is beyond 30 cholest units.

[0010](5) Rolled letter paper for tobacco given in any 1 paragraph of a (1) paragraph - (4) paragraph more than whose 50 mass % of said pulp is flax pulp.

(6) Rolled letter paper for tobacco given in any 1 paragraph of a (1) paragraph - (5) paragraph by which precipitated calcium carbonate of 10 - 40 mass % is contained in rolled letter paper for tobacco.

[0011]This invention includes the following inventions further.

(7) Rolled letter paper for tobacco given in any 1 paragraph of a (1) paragraph - (6) paragraph whose mean particle diameter of said goethite is 0.01-2.0 micrometers.

(8) Rolled letter paper for tobacco given in any 1 paragraph of a (1) paragraph - (7) paragraph whose BET specific surface area of said goethite is 0.2-200m<sup>2</sup>/g.

(9) Rolled letter paper for tobacco given in any 1 paragraph of a (1) paragraph - (8) paragraph which freeness of pulp is the freeness according to a Canada standard method (however, concentration of pulp suspension is made into 0.1%) by the wire method mentioned later, and is 100-200 ml.

[0012]

[Embodiment of the Invention]Usually, the rolled letter paper for tobacco used pulp and precipitated calcium carbonate as the main ingredients, and the combustion improver is contained so that there may be no going out, also when did not smoke tobacco and it is neglected. A sidestream smoke is smoke when not smoking tobacco, and when combustion temperature is low temperature, it is easy to generate it. Then, since oxygen will be supplied, the rate of combustion will increase and it will burn at an elevated temperature if tobacco is attracted, a sidestream smoke is hardly generated. Therefore, a sidestream smoke decreases by raising the rate of combustion.

[0013]The rolled letter paper for tobacco of this invention is the rolled letter paper for tobacco which reduced the sidestream smoke remarkably as compared with the conventional rolled letter paper for tobacco not affecting the smoking taste of tobacco but holding the moderate rate of combustion by making goethite contain. Goethite has the feature in not having an adverse effect on the smoking taste of tobacco at all, and does not affect the smoking taste of tobacco with the character, but makes possible the target of this invention of decreasing a sidestream smoke remarkably.

[0014]The goethite used by this invention is a particle with a mean particle diameter of 0.01-2.0 micrometers, and many long and slender detailed fiber particles arrange it regularly, and it forms spindle-shaped particles. Especially, a 0.1-1.0-micrometer particle is preferred from points, such as flammability, handling fitness, and a yield. The value of the desirable BET specific surface area of goethite is 0.2-200m<sup>2</sup>/g, and its 1-150-m<sup>2</sup>/g is more preferred.

[0015]The goethite used by this invention has a large BET specific surface area as mentioned above, It seems that it has a high catalysis which promotes combustion of combustibles, and it seems that the effect that this catalysis increases the combustion enhancement effect which cannot be attained in the alkali metal salt conventionally used as a combustion improver, and reduces a sidestream smoke is brought about.

[0016]The quantity of the goethite used for the rolled letter paper for tobacco of this invention is one to 10 mass part preferably 0.1 to 20 mass part to bone-dry pulp 100 mass part. Unless

content is less than 0.1 mass parts, a combustion facilitatory effect is not acquired, and when content exceeds 20 mass parts, coloring is conspicuous and there is a problem of burning too much.

[0017]In addition to goethite, the further reduction of a sidestream smoke of the rolled letter paper for tobacco of this invention is attained [ that it cannot attain when goethite or an alkali-metal-salt combustion improver is used independently, respectively, and ] by using together a publicly known alkali-metal-salt combustion improver conventionally. Although alkali metal salt, such as carbonic acid, formic acid, acetic acid, propionic acid, oxalic acid, malonic acid, succinic acid, glutaric acid, malic acid, lactic acid, citrate, and tartaric acid, is mentioned as an alkali-metal-salt combustion improver, When especially potassium citrate and sodium acid citrate are used as a combustion promotion additive, reduction in a sidestream smoke is remarkable and preferred.

[0018]The quantity of an alkali-metal-salt combustion improver is 1.0 to 3.0 mass part preferably 0.5 to 5.0 mass part to bone-dry pulp 100 mass part. If the quantity of an alkali-metal-salt combustion improver is less than a minimum, a combustion facilitatory effect is insufficient, and if a maximum is exceeded, a combustion facilitatory effect will decrease conversely. Internal [ of the addition to the rolled letter paper for tobacco of goethite and an alkali-metal-salt combustion improver ] may be carried out at the time of paper manufacture, and externally adding may be carried out by size press etc. after paper making.

[0019]Although the pulp in particular used for the rolled letter paper for tobacco of this invention is not limited, it is usable in seed fiber pulp, such as leaf fiber pulp, such as herb bast fiber pulp, such as broad-leaved tree wood pulp, needle-leaf tree wood pulp, a hemp, \*\*\*\*\* , a jute, and a kenaf, hemp of Manila, and a henequen, cotton, and linters, etc. However, flax pulp is preferred in respect of it being easy to secure permeability and tensile strength being made highly, and smoking taste being good and there being etc., and it is preferred that more than 50 mass % of the pulp used is flax pulp.

[0020]When the freeness of pulp at the time of paper making decreases a sidestream smoke, it is important. The freeness of the pulp used for the rolled letter paper for tobacco of this invention is the freeness according to the Canada standard method (however, concentration of pulp suspension is made into 0.1%.) by the wire method mentioned later, and its 100-200 ml is preferred. If it is in the tendency for tensile strength to be insufficient and for workability to worsen if freeness is less than a minimum and a maximum is exceeded, required permeability is not obtained and the reduction effect of a sidestream smoke is not enough.

[0021]Although there are precipitated calcium carbonate, titanium oxide, clay, kaolin, silica, zeolite, etc. which are usually well used as a loading material which are the other main ingredients, Although it takes out the opacity of paper, and it not only can maintain permeability, but the point of smoking taste to precipitated calcium carbonate is preferred and

the addition can be chosen arbitrarily, it is preferred 10-40 mass % That precipitated calcium carbonate is contained in the rolled letter paper for tobacco. If the quantity of precipitated calcium carbonate is less than 10 mass %, the permeability of the rolled letter paper for tobacco is low, and if 40 mass % is exceeded, it will become insufficient [ powder omission and tensile strength ].

[0022]The breathability of the rolled letter paper for tobacco affects the rate of combustion greatly. In the rolled letter paper for tobacco of this invention, it is preferred that the permeability of the completed rolled letter paper is beyond 30 cholest units, it is a 30 - 100 cholest unit more preferably, and is 30 - 80 cholest unit still more preferably. For a smoker, if permeability is not filled per 30 cholest, since flammability is inferior, the problem that the amount of nicotine tar increases not only arises, but it will stand and disappear or a sidestream smoke will increase due to the fall of the rate of combustion resulting from the shortage of breathability.

[0023]

[Example]Although an example explains this invention concretely below, this invention is not limited to these examples. In the following examples and comparative examples, a measuring method and the valuation method are as follows.

[0024]<Measurement of freeness> flax pulp and rolled-letter-paper timber material pulp have remarkably bad filterability as compared with usual paper pulp, and measurement of the freeness according to a Canada standard method is difficult. Then, it lowered to 0.1% from 0.3% to which the concentration of pulp slurry is specified by a Canada standard method, and the plate in which the small hole further attached to the \*\*\*\*\* lower part of a measuring instrument opened was changed into the mesh plate of 80 meshes, and was measured, and this value was made into CSF freeness. This measuring method is known for the paper-making industry as a 1g wire method.

[0025]Using the unit of the extract sheet created by the example and comparative example below <creation of a hand-rolled cigarette> and commercial tobacco (trade name; mild seven), the with 60 mm in length, the circumference of 25 mm, and a volume mass of 0.80\*\*0.02 g hand-rolled cigarette was created, and the following examinations were presented.

[0026]It was shown in the <physical properties of used goethite> table 1.

[0027]

[Table 1]

物性	値
成分	$\alpha$ -FeOOH
結晶構造	スピンドル
B E T 比表面積	86.3 $m^2/g$
水分	0.98%
p H	8.1
粒子径	0.25 $\mu m$

[0028]< permeability; it measured using product permeability meter ppmmade by cholest >  
U.S. FILTRONA 100.

Cholest unit; the air passing flow rate for 1 minute is expressed with  $\text{cm}^3$  per  $1\text{-cm}^2$  under a  $100\text{mmH}_2\text{O}$  condition.

[0029]Lit at the tip of the <amount of sidestream smokes> hand-rolled cigarette, and stand tobacco horizontally, it was made to combust spontaneously, and the quantity of the sidestream smoke which rises was observed visually. a judging standard -- O: -- little \*\*: -- quite large x: -- many was presupposed.

[0030]In the state of spontaneous combustion of the <rate-of-combustion> preceding clause, time for tobacco to burn 1 cm was measured in the second. The desirable rate of combustion is 100 to 200 seconds/cm.

[0031]Beating of the bleached [ example 1 ] flax pulp was carried out to 150 ml of CSF freeness, goethite (made by Toda Kogyo Corp.) and precipitated calcium carbonate (PCX850 Shiraishi industrial company make) were added, the sheet of U.S. tsubo  $30\text{ g/m}^2$  was created with the TAPPI normalized form extract machine, and each examination was presented. The loadings to bone-dry pulp 100 mass part of goethite were made into 1.0 mass parts. The compounding rate in the rolled letter paper for tobacco of calcium carbonate was made into 30 mass parts.

[0032]Except having made the loadings of example 2 goethite into 2.0 mass parts, the extract sheet was created like Example 1 and each examination was presented.

[0033]0.5 mass parts of potassium citrate were further applied to the sheet of example 3 Example 1 to sheet bone-dry pulp 100 mass part, the extract sheet of Example 3 was created, and each examination was presented.

[0034]Except the CSF freeness of example 4 flax pulp having been 200 ml, the extract sheet was created like Example 1 and each examination was presented.

[0035]Except having made the loadings of example 5 goethite into 0.01 mass parts, the extract sheet was created like Example 1 and each examination was presented.

[0036]Except having made the compounding rate of example 6 goethite into 25 mass parts, the extract sheet was created like Example 1 and each examination was presented. This sample had coloring.

[0037]Except not making comparative example 1 goethite contain, the extract sheet was created like Example 1 and it was considered as the sheet for comparison. Fire stands, and cannot go out and smoke.

[0038]1.0 mass parts of potassium citrate were applied to the sheet which does not contain the goethite of the comparative example 2 comparative example 1 to sheet bone-dry pulp 100 mass part, and it was considered as the sheet for comparison. Since the rate of combustion is

slow, there are many sidestream smokes.

[0039]The test result of each of said example is shown in Table 2.

[Table 2]

区分	燃焼促進剤添加量	C S F ろ水度 (mL)	通気度 (CU)	副流煙量	燃焼速度 (秒/cm)
実施例 1	ゲータイト 1.0 質量部	150	44	○	165
実施例 2	ゲータイト 2.0 質量部	150	42	○	130
実施例 3	ゲータイト 1.0 質量部 クエン酸カリウム 0.5 質量部	150	44	○	140
実施例 4	ゲータイト 1.0 質量部	200	30	○	150
実施例 5	ゲータイト 0.01 質量部	150	44	△	200
実施例 6	ゲータイト 2.5 質量部	150	46	○	90
比較例 1	無し	150	45	×	立ち消える
比較例 2	ゲータイト 無し クエン酸カリウム 1.0 質量部	150	44	△	210

[0040]

[Effect of the Invention]In the example of this invention, compared with the comparative example, the rate of combustion required as rolled letter paper for tobacco was held, and it became possible to reduce the amount of sidestream smokes so that clearly from Table 2. The rolled letter paper for tobacco of this invention is outstanding rolled letter paper for tobacco which does not affect the smoking taste of tobacco and does not raise a manufacturing cost, either.

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**TECHNICAL FIELD**

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[Field of the Invention] This invention relates to the rolled letter paper for tobacco for manufacturing tobacco with few sidestream smokes.

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**PRIOR ART**

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[Description of the Prior Art]If the mainstream smoke attracted by a smoker through a tobacco main part and fire leave just on the occasion of smoking of tobacco, the sidestream smoke which rises from this tip will occur, but a sidestream smoke also causes a health issue at the same time it gives the surrounding nonsmoker displeasure. For this reason, it ranks with not affecting smoking taste, having the moderate rate of combustion, that the leaf of a cigarette is transparent and is not seen (opacity), self-winding fitness (tensile strength), etc. as the required characteristic of the rolled letter paper for tobacco, and it is becoming still more important to decrease a sidestream smoke in recent years.

[0003]About decreasing the sidestream smoke of tobacco, conventionally Then, JP,48-9000,A, JP,48-61699,A, JP,58-183082,A, JP,63-87967,A, JP,2-99699,A, JP,2-501075,A, JP,3-33299,A, JP,3-43068,A, JP,3-151868,A, Many methods are proposed by gazettes, such as JP,3-180597,A, JP,3-500553,A, JP,5-236929,A, JP,5-324397,A, JP,10-81,A, and JP,11-124800,A.

[0004]For example, to JP,5-236929,A, in order to decrease a sidestream smoke, making the rolled letter paper for tobacco contain carbon is indicated.

In JP,10-81,A, in order to decrease a sidestream smoke, the rolled letter paper for tobacco using pulp of the high degree of beating is indicated.

To JP,11-124800,A, in order to mask the bad smell of a sidestream smoke, making lactone contain is indicated. although that which surely is effective is accepted, a manufacturing method is difficult, these proposals have many problems, impairing the smoking taste of tobacco or becoming a cost hike, and it is actual that the yet sufficiently satisfying rolled letter paper for tobacco is not found out.

[0005]By using alkali metal salt especially potassium citrate, and sodium acid citrate for the rolled letter paper for tobacco as a combustion promotion additive, it is known that a sidestream smoke will decrease and, generally it is used (U.S. Pat. No. 4321377 specification).

However, although it is possible for the rate of combustion to become quick and to decrease a sidestream smoke to some extent as a result by adding alkali metal salt, there is evil in which regulation is difficult, the rate of combustion will fall rather if alkali metal salt is added superfluously, or smoking taste falls. Therefore, it is difficult to decrease a sidestream smoke, without changing smoking taste only by addition of alkali metal salt.

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**EFFECT OF THE INVENTION**

---

[Effect of the Invention]In the example of this invention, compared with the comparative example, the rate of combustion required as rolled letter paper for tobacco was held, and it became possible to reduce the amount of sidestream smokes so that clearly from Table 2. The rolled letter paper for tobacco of this invention is outstanding rolled letter paper for tobacco which does not affect the smoking taste of tobacco and does not raise a manufacturing cost, either.

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**TECHNICAL PROBLEM**

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[Problem(s) to be Solved by the Invention] An object of this invention is to provide the rolled letter paper for tobacco which decreased in number the sidestream smoke remarkably as compared with the conventional rolled letter paper for tobacco, not affecting the smoking taste of tobacco but holding the moderate rate of combustion.

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**MEANS**

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[Means for Solving the Problem] A result of this invention persons having investigated a new combustion promotion additive, and having considered application to rolled letter paper for tobacco, Without changing an effect which cannot be attained if the above-mentioned alkali metal salt was only used as a combustion promotion additive, i.e., smoking taste, a substance to which a sidestream smoke is reduced is found out and it came to complete this invention.

[0008] This invention for solving the above-mentioned technical problem includes the following inventions.

(1) Rolled letter paper for tobacco using pulp and precipitated calcium carbonate as the main ingredients, and containing goethite further.

(2) Rolled letter paper for tobacco which contains goethite (alpha-FeOOH) of 0.1 to 20 mass part to bone-dry pulp 100 mass part in rolled letter paper for tobacco which uses pulp and precipitated calcium carbonate as the main ingredients.

[0009](3) (1) which contains an alkali-metal-salt combustion improver of 0.5 - 5.0 mass part to bone-dry pulp 100 mass part further, or rolled letter paper for tobacco given in (2) paragraphs.

(4) Rolled letter paper for tobacco given in any 1 paragraph of a (1) paragraph - (3) paragraph whose permeability is beyond 30 cholest units.

[0010](5) Rolled letter paper for tobacco given in any 1 paragraph of a (1) paragraph - (4) paragraph more than whose 50 mass % of said pulp is flax pulp.

(6) Rolled letter paper for tobacco given in any 1 paragraph of a (1) paragraph - (5) paragraph by which precipitated calcium carbonate of 10 - 40 mass % is contained in rolled letter paper for tobacco.

[0011] This invention includes the following inventions further.

(7) Rolled letter paper for tobacco given in any 1 paragraph of a (1) paragraph - (6) paragraph whose mean particle diameter of said goethite is 0.01-2.0 micrometers.

(8) Rolled letter paper for tobacco given in any 1 paragraph of a (1) paragraph - (7) paragraph

whose BET specific surface area of said goethite is 0.2-200m<sup>2</sup>/g.

(9) Rolled letter paper for tobacco given in any 1 paragraph of a (1) paragraph - (8) paragraph which freeness of pulp is the freeness according to a Canada standard method (however, concentration of pulp suspension is made into 0.1%) by the wire method mentioned later, and is 100-200 ml.

[0012]

[Embodiment of the Invention]Usually, the rolled letter paper for tobacco used pulp and precipitated calcium carbonate as the main ingredients, and the combustion improver is contained so that there may be no going out, also when did not smoke tobacco and it is neglected. A sidestream smoke is smoke when not smoking tobacco, and when combustion temperature is low temperature, it is easy to generate it. Then, since oxygen will be supplied, the rate of combustion will increase and it will burn at an elevated temperature if tobacco is attracted, a sidestream smoke is hardly generated. Therefore, a sidestream smoke decreases by raising the rate of combustion.

[0013]The rolled letter paper for tobacco of this invention is the rolled letter paper for tobacco which reduced the sidestream smoke remarkably as compared with the conventional rolled letter paper for tobacco not affecting the smoking taste of tobacco but holding the moderate rate of combustion by making goethite contain. Goethite has the feature in not having an adverse effect on the smoking taste of tobacco at all, and does not affect the smoking taste of tobacco with the character, but makes possible the target of this invention of decreasing a sidestream smoke remarkably.

[0014]The goethite used by this invention is a particle with a mean particle diameter of 0.01-2.0 micrometers, and many long and slender detailed fiber particles arrange it regularly, and it forms spindle-shaped particles. Especially, a 0.1-1.0-micrometer particle is preferred from points, such as flammability, handling fitness, and a yield. The value of the desirable BET specific surface area of goethite is 0.2-200m<sup>2</sup>/g, and its 1-150-m<sup>2</sup>/g is more preferred.

[0015]The goethite used by this invention has a large BET specific surface area as mentioned above, It seems that it has a high catalysis which promotes combustion of combustibles, and it seems that the effect that this catalysis increases the combustion enhancement effect which cannot be attained in the alkali metal salt conventionally used as a combustion improver, and reduces a sidestream smoke is brought about.

[0016]The quantity of the goethite used for the rolled letter paper for tobacco of this invention is one to 10 mass part preferably 0.1 to 20 mass part to bone-dry pulp 100 mass part. Unless content is less than 0.1 mass parts, a combustion facilitatory effect is not acquired, and when content exceeds 20 mass parts, coloring is conspicuous and there is a problem of burning too much.

[0017]In addition to goethite, the further reduction of a sidestream smoke of the rolled letter

paper for tobacco of this invention is attained [ that it cannot attain when goethite or an alkali-metal-salt combustion improver is used independently, respectively, and ] by using together a publicly known alkali-metal-salt combustion improver conventionally. Although alkali metal salt, such as carbonic acid, formic acid, acetic acid, propionic acid, oxalic acid, malonic acid, succinic acid, glutaric acid, malic acid, lactic acid, citrate, and tartaric acid, is mentioned as an alkali-metal-salt combustion improver, When especially potassium citrate and sodium acid citrate are used as a combustion promotion additive, reduction in a sidestream smoke is remarkable and preferred.

[0018]The quantity of an alkali-metal-salt combustion improver is 1.0 to 3.0 mass part preferably 0.5 to 5.0 mass part to bone-dry pulp 100 mass part. If the quantity of an alkali-metal-salt combustion improver is less than a minimum, a combustion facilitatory effect is insufficient, and if a maximum is exceeded, a combustion facilitatory effect will decrease conversely. Internal [ of the addition to the rolled letter paper for tobacco of goethite and an alkali-metal-salt combustion improver ] may be carried out at the time of paper manufacture, and externally adding may be carried out by size press etc. after paper making.

[0019]Although the pulp in particular used for the rolled letter paper for tobacco of this invention is not limited, it is usable in seed fiber pulp, such as leaf fiber pulp, such as herb bast fiber pulp, such as broad-leaved tree wood pulp, needle-leaf tree wood pulp, a hemp, \*\*\*\*\* , a jute, and a kenaf, hemp of Manila, and a henequen, cotton, and linters, etc. However, flax pulp is preferred in respect of it being easy to secure permeability and tensile strength being made highly, and smoking taste being good and there being etc., and it is preferred that more than 50 mass % of the pulp used is flax pulp.

[0020]When the freeness of pulp at the time of paper making decreases a sidestream smoke, it is important. The freeness of the pulp used for the rolled letter paper for tobacco of this invention is the freeness according to the Canada standard method (however, concentration of pulp suspension is made into 0.1%.) by the wire method mentioned later, and its 100-200 ml is preferred. If it is in the tendency for tensile strength to be insufficient and for workability to worsen if freeness is less than a minimum and a maximum is exceeded, required permeability is not obtained and the reduction effect of a sidestream smoke is not enough.

[0021]Although there are precipitated calcium carbonate, titanium oxide, clay, kaolin, silica, zeolite, etc. which are usually well used as a loading material which are the other main ingredients, Although it takes out the opacity of paper, and it not only can maintain permeability, but the point of smoking taste to precipitated calcium carbonate is preferred and the addition can be chosen arbitrarily, it is preferred 10-40 mass % That precipitated calcium carbonate is contained in the rolled letter paper for tobacco. If the quantity of precipitated calcium carbonate is less than 10 mass %, the permeability of the rolled letter paper for tobacco is low, and if 40 mass % is exceeded, it will become insufficient [ powder omission and

tensile strength ].

[0022]The breathability of the rolled letter paper for tobacco affects the rate of combustion greatly. In the rolled letter paper for tobacco of this invention, it is preferred that the permeability of the completed rolled letter paper is beyond 30 cholest units, it is a 30 - 100 cholest unit more preferably, and is 30 - 80 cholest unit still more preferably. For a smoker, if permeability is not filled per 30 cholest, since flammability is inferior, the problem that the amount of nicotine tar increases not only arises, but it will stand and disappear or a sidestream smoke will increase due to the fall of the rate of combustion resulting from the shortage of breathability.

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[Translation done.]

**\* NOTICES \***

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- 1.This document has been translated by computer. So the translation may not reflect the original precisely.
- 2.\*\*\*\* shows the word which can not be translated.
- 3.In the drawings, any words are not translated.

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**EXAMPLE**

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[Example]Although an example explains this invention concretely below, this invention is not limited to these examples. In the following examples and comparative examples, a measuring method and the valuation method are as follows.

[0024]<Measurement of freeness> flax pulp and rolled-letter-paper timber material pulp have remarkably bad filterability as compared with usual paper pulp, and measurement of the freeness according to a Canada standard method is difficult. Then, it lowered to 0.1% from 0.3% to which the concentration of pulp slurry is specified by a Canada standard method, and the plate in which the small hole further attached to the \*\*\*\*\* lower part of a measuring instrument opened was changed into the mesh plate of 80 meshes, and was measured, and this value was made into CSF freeness. This measuring method is known for the paper-making industry as a 1g wire method.

[0025]Using the unit of the extract sheet created by the example and comparative example below <creation of a hand-rolled cigarette> and commercial tobacco (trade name; mild seven), the with 60 mm in length, the circumference of 25 mm, and a volume mass of 0.80\*\*0.02 g hand-rolled cigarette was created, and the following examinations were presented.

[0026]It was shown in the <physical properties of used goethite> table 1.

[0027]

[Table 1]

物性	値
成分	$\alpha$ -FeOOH
結晶構造	スピンドル
BET比表面積	86.3 m <sup>2</sup> /g
水分	0.98%
pH	8.1
粒子径	0.25 μm

[0028]< permeability; it measured using product permeability meter ppmmade by cholest >  
U.S. FILTRONA 100.

Cholest unit; the air passing flow rate for 1 minute is expressed with  $\text{cm}^3$  per  $1\text{-cm}^2$  under a  $100\text{mmH}_2\text{O}$  condition.

[0029] Lit at the tip of the <amount of sidestream smokes> hand-rolled cigarette, and stand tobacco horizontally, it was made to combust spontaneously, and the quantity of the sidestream smoke which rises was observed visually. a judging standard -- O: -- little \*\*: -- quite large x: -- many was presupposed.

[0030] In the state of spontaneous combustion of the <rate-of-combustion> preceding clause, time for tobacco to burn 1 cm was measured in the second. The desirable rate of combustion is 100 to 200 seconds/cm.

[0031] Beating of the bleached [ example 1 ] flax pulp was carried out to 150 ml of CSF freeness, goethite (made by Toda Kogyo Corp.) and precipitated calcium carbonate (PCX850 Shiraishi industrial company make) were added, the sheet of U.S. tsubo  $30\text{ g/m}^2$  was created with the TAPPI normalized form extract machine, and each examination was presented. The loadings to bone-dry pulp 100 mass part of goethite were made into 1.0 mass parts. The compounding rate in the rolled letter paper for tobacco of calcium carbonate was made into 30 mass parts.

[0032] Except having made the loadings of example 2 goethite into 2.0 mass parts, the extract sheet was created like Example 1 and each examination was presented.

[0033] 0.5 mass parts of potassium citrate were further applied to the sheet of example 3 Example 1 to sheet bone-dry pulp 100 mass part, the extract sheet of Example 3 was created, and each examination was presented.

[0034] Except the CSF freeness of example 4 flax pulp having been 200 ml, the extract sheet was created like Example 1 and each examination was presented.

[0035] Except having made the loadings of example 5 goethite into 0.01 mass parts, the extract sheet was created like Example 1 and each examination was presented.

[0036] Except having made the compounding rate of example 6 goethite into 25 mass parts, the extract sheet was created like Example 1 and each examination was presented. This sample had coloring.

[0037] Except not making comparative example 1 goethite contain, the extract sheet was created like Example 1 and it was considered as the sheet for comparison. Fire stands, and cannot go out and smoke.

[0038] 1.0 mass parts of potassium citrate were applied to the sheet which does not contain the goethite of the comparative example 2 comparative example 1 to sheet bone-dry pulp 100 mass part, and it was considered as the sheet for comparison. Since the rate of combustion is slow, there are many sidestream smokes.

[0039] The test result of each of said example is shown in Table 2.

[Table 2]

区分	燃焼促進剤添加量	C S F ろ水度 (ml)	通気度 (CU)	副流煙量 (秒/cm)	燃焼速度 (秒/cm)
実施例 1	ゲータイト 1.0 質量部	150	44	○	165
実施例 2	ゲータイト 2.0 質量部	150	42	○	130
実施例 3	ゲータイト 1.0 質量部 クエン酸カリウム 0.5 質量部	150	44	○	140
実施例 4	ゲータイト 1.0 質量部	200	30	○	150
実施例 5	ゲータイト 0.01 質量部	150	44	△	200
実施例 6	ゲータイト 2.5 質量部	150	46	○	90
比較例 1	無し	150	45	×	立ち消える
比較例 2	ゲータイト 無し クエン酸カリウム 1.0 質量部	150	44	△	210

[Translation done.]